Effect of Martensite Transformation on Toughness of 0.12 C-0.5Ti-9Ni-18Cr Stainless Steel

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***Abstract*- The paper describes the effect of martensitic transformation on toughness for a particular grade of stainless steel. An experimental study was carried out to understand austenite-martensite transformation in this steel and their correlation with toughness at various temperatures ranging from 303 K to 103 K. The transformed martensite has adverse reduced impact toughness of steel. This paper highlights the details of experiment carried out and brings out effect of transformed martensite on toughness of the steel especially at a temperature range up to 100 K. It was reported that deformation of 0.12C-0.5Ti-9Ni-18Cr-2Mn stainless steel at 77 K enhanced the kinetics of austenite-martensite transformation.**

Keywords- Stainless Steel; Transformed Martensite; Austenite-Martensite Transformation; Deformation of Austenite Stainless Steel at Cryo Tempearture

I. INTRODUCTION

Austenitic stainless steels are being widely used for a variety of components in propulsion system, which demands good formability, high corrosion resistance and excellent toughness at cryogenic temperatures.

A liquid hydrogen conduit pipeline that transfer cryogenic fuel from its storage tank to the output collector of a propulsion system developed cracks at bends while undergoing a vibration test. The tube, made of 0.12C-0.5Ti-9Ni-18Cr-2Mn grade stainless was bent using frozen water (ice) at 77 K. The metallurgical investigation carried out on cracked tube indicated presence of transformed martensite at bent region. Interior surface of the tube had multiple fine longitudinal cracks, resulted from broken die used during extrusion of tube. The details of investigation and causes for such cracking are reported elsewhere [1].

This steel retains austenitic phase at room temperature (RT). However, deformation induced austenite-martensite transformation; especially at temperature below RT is well understood. The phenomenon of austenite-martensite transformation and its effect on notch sensitivity is well researched and documented. However, its effect on the impact toughness in this steel is scares. An experimental study was carried out to understand austenite-martensite transformation in this steel and their correlation with toughness at various temperatures ranging from 303 K to 103 K.

The aims of the experimental studies were:

i) to bend 0.12C-0.5Ti-9Ni-18Cr tube using frozen water (ice) at 77 K and evaluation of transformed martensite, this was planned to simulate the condition of pipeline in propulsion system, which developed cracking;

ii) to impart different degree of working at ambient as well as 77 K and to establish relation between transformed martensite resulted from austenite-martensite transformation, degree of deformation and temperatures;

iii) to evaluate the effect of martensite on toughness of steel, at temperatures ranging from ambient to 100 K.

II. MATERIAL AND EXPERIMENTAL PROCEDURE

0.12C-0.5Ti-9Ni-18Cr-2Mn stainless steel of size 40 mm OD and 2 mm thickness was used for tube bending, while slices cut from 80 mm diameter rod was taken for imparting various degree of deformation at a range of cryo temperatures.

Experiments were carried out in various steps given below.

A. 40 mm diameter and 2 mm thick tube was filled with water and immersed in liquid nitrogen for an hour enabling water to freeze within inside. Tube with frozen ice inside was bent through 90°. Frozen ice acted as a mandrel and thus resulted in wrinkle free bent tube. The bent tube is shown in Figure 1. Austenite transformed to martensite depending on the degree of deformation. Martensite (α –phase) was measured at a number of locations on straight tube and bent region.

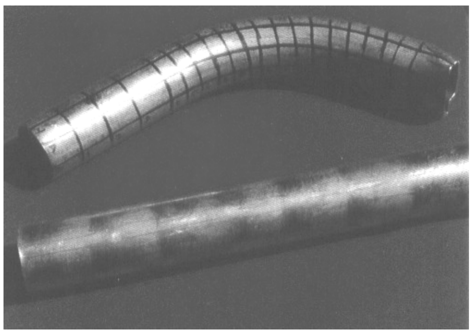

Fig. 1 Frozen water filled tube after bending

B. In an another attempt, slices were cut from 80 mm diameter rod and subjected to various treatment as follows;

   a) exposed to 77 K for 12, 24 and 48 hours, and measuring the quantity of the transformed α –phase;
   b) imparted 5, 10, 15, 20, 25 and 35% deformation at room temperature and quantification of α -phase;
   c) imparted 5, 10, 15, 20, 25 and 35% deformation at ambient temperature, cooled to 77K for 48 hours and evaluation for α – phase;
   d) imparted 10 and 20% deformation at 77 K and evaluating α- phase.

C. Study the effect of transformed martensite on impact toughness of the steel. Impact toughness by Charpy Impact testing was evaluated at ambient, 193 K, 163 K, 133 K, and 103 K for the material in the following condition:

   a) samples containing no α- phase (i.e. samples in ‘as received condition’);
   b) steel containing known amount of transformed α- phase, resulted from austenite – martensite transformation.

Specimens for impact toughness evaluation were sliced off from the material which had undergone austenite-martensite transformation due to deformation at cryo temperature and had a range of transformed martensite due to variation in extent of deformation. Specimens were segregated in batches such that a particular batch of specimen contained same amount of transformed martensite.

During impact test, thermocouple was fixed on each specimen using adhesive aluminum foil, to measure the exact temperature at the time of fracture.

D. During impact toughness test, the material at the vicinity of notch was expected to undergo severe deformation under dynamic loading and hence further austenite to martensite transformation occurred. To understand this, specimens with 0% and 30% transformed martensite were evaluated for martensite content at the notch/fracture end before and after the impact test.

III. RESULTS AND DISCUSSIONS

The amount of transformed martensite was determined using a feritoscope. Average of the two measurements on each location is reported.

The initial material used in this experimental study was 40 mm OD and 2 mm thick tube for bending at cryo-temperature and slices of 80 mm diameter rod for imparting different amount of deformation at various temperatures.

The martensite (α-phase) content of rod and tube in as received condition was as low as 0% and 2% respectively. This indicated material is predominantly in austenitic condition. The tube was bent using frozen ice and martensite content measured at different locations is given in Table I.

A schematic sketch of the locations ‘A’ through ‘H’ along the circumference of the tube and locations ‘01’ through ‘18’ on the surface and along the axis of the bent tube are shown in Figure 2. The bent region of the tube on outer axis (i.e. location 9-A) had maximum 20% martensite, while region of neutral axis at bent region (i.e. locations 9-C and 9- G) had reasonably less transformed martensite.

TABLE I. MARTENSITE CONTENT OF COLD DEFORMED TUBE (BENT THROUGH 90° WITH FROZEN WATER INSIDE THE TUBE)

Cross section no.	A	B	C	D	E	F	G	H
1	-	-	0.27	0.35	0.30	0.24	-	-
2	-	-	0.55	-	0.20	0.18	-	-
3	-	-	0.21	-	-	-	-	-
4	0.27	0.26	0.25	0.19	0.23	-	0.30	0.30
5	1.6	0.80	0.24	0.28	0.34	0.32	-	0.72
6	6.6	3.1	0.39	0.49	1.2	0.54	0.34	4.9
7	14.4	10.0	0.73	0.68	2.7	1.1	0.27	11.0
8	16.6	13.4	0.77	1.1	5.0	2.0	0.54	13.9
9	20.0	14.4	0.90	2.0	7.5	2.8	1.0	16.3
10	17.5	13.1	0.96	2.5	8.6	3.1	1.1	15.0
11	12.8	11.8	1.1	2.7	8.7	2.9	0.98	10.3
12	3.7	11.2	0.90	1.7	7.9	2.6	1.2	4.8
13	2.7	7.9	1.3	1.3	5.8	2.2	1.3	4.2
14	2.9	6.7	0.82	1.4	4.8	1.6	1.2	4.5
15	2.2	5.1	0.74	1.4	4.4	1.7	1.3	2.5
16	1.1	2.7	1.2	1.6	3.4	1.9	0.69	0.76
17	0.56	0.39	0.88	0.46	0.78	1.5	0.56	0.60
18	0.32	-	0.49	0.23	-	0.37	0.80	0.29

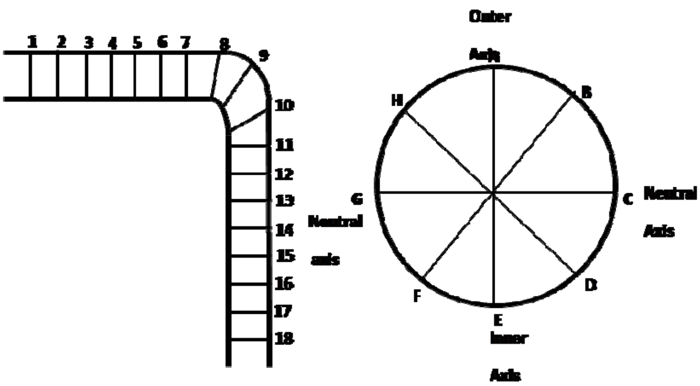

Fig. 2 Schematic sketch indicating locations on the bend tube where α- phase was evaluated.

The slices taken out from 80 mm diameter rod and exposed to 77K for 12, 24 and 48 hours showed maximum 0.1% martensite (Table II). This indicated that merely cooling the steel to cryo temperature did not transform austenite to martensite. Imparting deformation up to 35% at room temperature (RT) had marginal increase in martensite content (Table III), however kinetic was slow enough to result in 1.1% transformed martensite with 35% deformation. Even cooling theses blocks to 77K in liquid nitrogen showed no increases in martensite content (Table IV).

TABLE II MARTENSITE CONTENT ON COOLING TO LN_2 TEMPERATURE (77K)

Time (hours)	0	12	24	48
% Martensite	0.0	0.1	0.1	0.1

TABLE III MARTENSITE CONTENT AFTER DEFORMATION AT ROOM TEMPERATURE

% Reduction	0	5	10	15	20	25	35
% Martensite	0.0	0.0	0.18	0.23	0.31	0.42	1.10

TABLE IV MARTENSITE CONTENT AFTER ROOM TEMPERATURE DEFORMATION FOLLOWED BY HOLDING IN LN_2 TEMPERATURE (77K)

%Reduction	0	5	10	15	20	25	35
% Martensite after 30 mts	0	0	0.20	0.21	0.32	0.36	1.1
% Martensite after 24 hrs	0	0.1	0.22	0.24	0.32	0.39	1.12
% Martensite after 48 hrs	0	0.1	0.23	0.24	0.33	0.39	1.2

Deformation (10% and 20%) at liquid nitrogen temperature (77K) could transform austenite to martensite and at certain location, transformed martensite up to 50% was seen (Table V). This has confirmed that the kinetics of austenite-martensite transformation was controlled by the temperature at which deformation took place and the extent of deformation. The variation in the martensite content i.e. 10%-20% martensite for 10 % deformation and 35%-50% martensite for 20% deformation (Table V) was attributed to non uniform deformation throughout the bulk of material. The top and bottom layers were deformed more severely than that of bulk material.

TABLE V MARTENSITE CONTENT WITH DIFFERENT AMOUNT OF DEFORMATION AT 77K

Deformation at 77K	10 %	20%
% Martensite	20 - 35 %	35 - 50 %

The deformed blocks were further sliced off (10 mm thickness) for impact test specimen fabrication. The specimens were grouped with respect to their martensite content. A set of specimen with 30% transformed martensite was evaluated for impact toughness at temperatures ranging from RT to 100 K. Similar tests were carried out for the specimens with 'nil' transformed martensite. Figure 3 shows the impact toughness of material with 'nil' martensite and '30%' martensite at various temperature of testing within the range of RT to 100 K. The drastic decrease in impact toughness was attributed to the presence of transformed martensite.

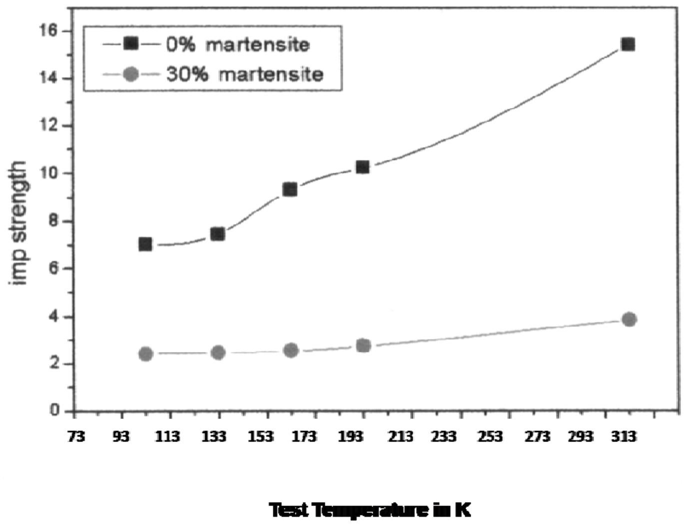

Fig. 3 Impact strength of steel with 30% martensite and without martensite

Further, few more specimens taken out from the blocks, deformed at 77K and had martensite content up to 51% were evaluated for impact toughness at 163K and 133K (Fig. 4). Result indicated that for a particular martensite content, impact toughness decreases with temperature.

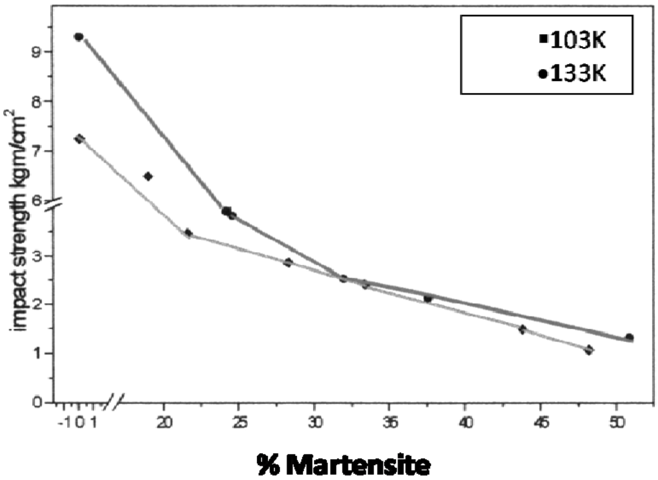

Fig. 4 Impact strength as a function of martensite content at different temperatures (163K and 103K)

Impact loading during toughness evaluation causes sudden localized deformation at and near notch region before fracture occurs. The effect of such severe deformation under dynamic loading to austenite-martensite transformation was studied. The specimens with 0% and 30% martensite were evaluated for martensite content at the fracture end after impact toughness test. Observations (Table VI) indicated that there was an increase in martensite content at the fracture end after the impact test due to severe deformation under dynamic loading. Difference was more predominant in the specimen containing no martensite initially. The specimens containing 30% initial martensite i.e. before the test could indicate marginal increase of 7% in martensite after the test. This was attributed to the severe deformation under dynamic loading at the notch site during the test.

TABLE VI VARIATION OF MARTENSITE BEFORE AND AFTER THE IMPACT TESTING

martensite content in % (before test)	Martensite content after impact test at				
	303K	193K	163K	133K	103K
0	1	18	23	28	31
30	36	37	37	37	37

Scanning Electron Microscopy (SEM) of the fracture surface of impact specimens with different martensite content, tested at various temperatures was carried out using PHILIPS XL 30 make scanning electron microscope (SEM). All the specimens revealed predominantly dimple mode of failure. This indicated that the steel even with lower impact strength, behaved in ductile mode. Two typical fractographs are shown in Figure 5.

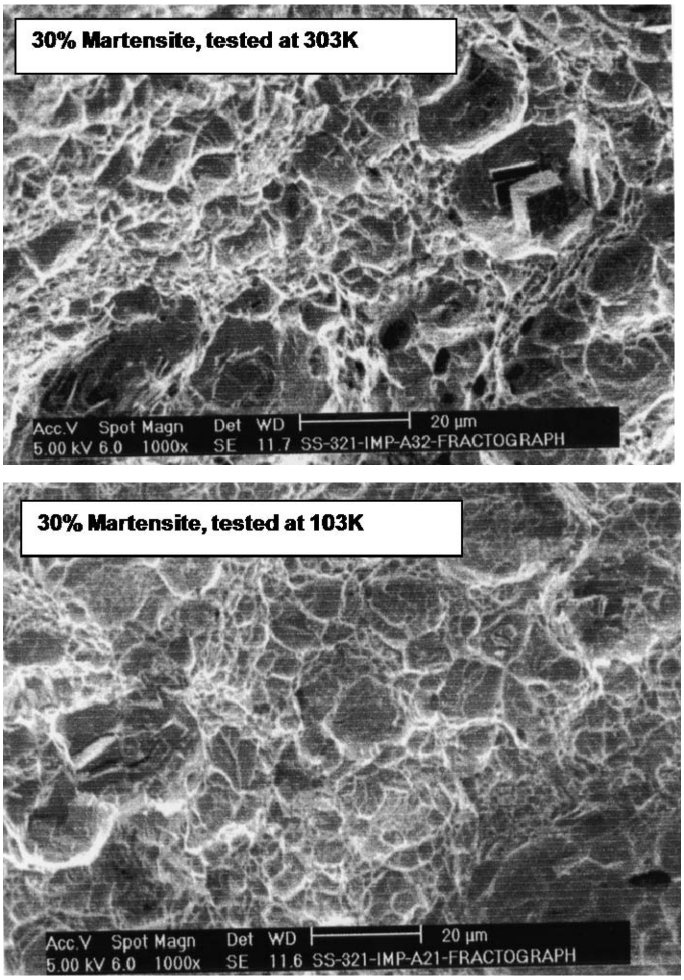

Fig. 5 SEM fractographs showing dimple mode of failure

IV. DISCUSSION

Austenite to martensite transformation is attributed to the chemical free energy change of martensite at lower temperature. Schematic illustration showing chemical free energies of austenite and martensite as a function of temperature is shown in Figure 6.

T_0 is the temperature at which the austenite and martensite are in equilibrium, and M_s is the temperature at which the transformation starts upon cooling. The difference in free energies between austenite and martensite transformation $\Delta G_{Ms}{}^{\gamma \rightarrow \acute{\alpha}}$ at M_s is the critical driving force for the onset of the martensite transformation. When the stress is applied to the austenite at temperature T_1 (between M_s and T_0), the mechanical driving force (U) due to stress is added to the chemical driving force and martensite transformation starts at the critical stress where the total driving force is equal to $\Delta G_{Ms}{}^{\gamma \rightarrow \acute{\alpha}}$. The critical mechanical driving force necessary for the stress induced martensitic transformation at temperature T_1 is given by the expression U' = ($\Delta G_{Ms}{}^{\gamma \rightarrow \acute{\alpha}}$ - $\Delta G_{T1}{}^{\gamma \rightarrow \acute{\alpha}}$). When a martensitic transformation starts by stressing a polycrystalline austenite in which the orientation of each grain is distributed at random, a martensite plate oriented in a particular plane and yields a maximum value of 'U' will be formed first [2].

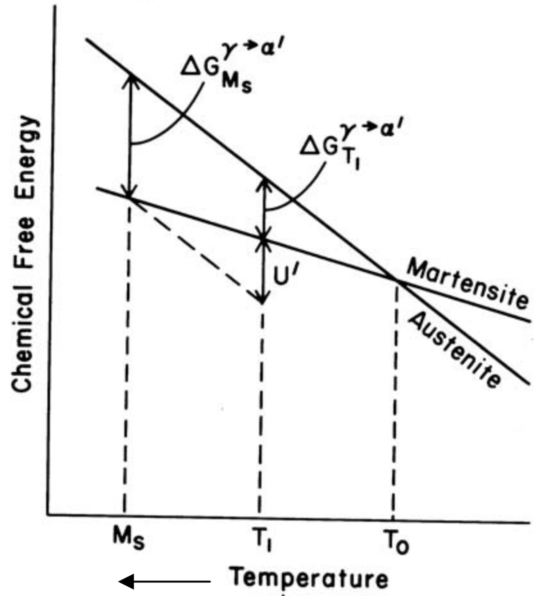

Fig. 6 Schematic illustration showing chemical free energies of austenite and martensite as a function of temperature

In the present experimental condition, exposing the tube and rod samples of 0.12C-0.5Ti-9Ni-18Cr stainless steel to 77K could not induce martensitic transformation, indicating that the Ms temperature was below -196°C. Samples of rod, which were deformed to 5, 10, 15, 20, 25 and 35% by compression at RT followed by cooling to 77K and homogenizing at this temperature for 48 hours, could not induce martensitic transformation. These results showed that room temperature deformation contributed to a very small increase in martensite content. Even after 35% reduction, only 1% martensite was formed. Holding steel alone at 77 K for 48 hours did not result in martensitic transformation. Even holding the steel after cold deformation (at RT) in liquid nitrogen (77 K) did not indicate any increase in martensite content as evidenced from Table 4.

The rod samples subjected to 10% and 20% deformation at 77 K itself, caused considerable amount of martensitic transformation. Bending of the tube with water frozen with LN_2, i.e. stressing the tube at 77 K, produced 20% martensite on the maximum bend region, while at neutral axis of tube,

where deformation was practically nil, it was bare minimum. This is an indication of start of martensitic transformation under influence of stress at temperature above Ms temperature. It is to be noted here that the mechanical driving force is provided by the deformation at lower temperature. Variation of martensite within the bend region is attributed to difference in the extent of strain during bending due to geometry of tube. Similar observations were made on samples from the rod subjected to deformation at liquid nitrogen temperature. The rod samples, which have undergone to 10% and 20% deformation also, indicated variation in the quantity of martesite transformed from place to place within the test block. This was attributed to the variation in the degree of deformation under forge press.

On impact, specimen deforms elastically until yielding takes place (plastic deformation), and a plastic zone develops at notch. As the test specimen continues to be deformed by impact, the plastic zone work hardens. This increases the stress and strain in the plastic zone until specimen fractures. The Charpy impact energy therefore includes the elastic strain energy, the plastic work done during yielding and the work done to create the fracture surface.

Since the Charpy impact energy comprises mostly of the plastic work of yielding of the specimen, it is affected by factors which change the yield behaviour of the material, such as temperature and the strain rate, through their effect on the behaviour of dislocations.

The impact toughness values at different test temperature indicated decrease in toughness at lower temperatures. This trend is seen for the rod sample in austenitic condition without any transformed martensite. The drop of toughness for the specimen with no martensite, from 15.4 Kgm/cm^2 at room temperature to 7.0 Kgm/cm^2 at 103K indicated a drop of 55% in toughness, whereas for the sample with 30% transformed martensite, the drop of impact toughness value from 3.90 Kgm/cm^2 at room temperature to 2.40 Kgm/cm^2 at 103 K, indicated a drop of 37% (Figure 4). Decrease in impact toughness with increase in martensite content was observed even at RT as well as at subzero temperatures. Specimens with 30% martensite showed 75% drop (from 15.3 Kgm/cm^2 to 3.8 Kgm/cm^2) in toughness at RT when compared with specimens having nil martensite. Similar drop of toughness was estimated to be 65% (from 7.1 Kgm/cm^2 to 2.4 Kgm/cm^2) at 103K (Figure 4).

The decrease in impact toughness with decrease in temperature was found to be gradual and was not sudden as exhibited by a material undergoing Ductile to Brittle transition (DBTT). The fractographs observed on the impact-tested specimen at different sub zero temperatures indicated ductile mode of failure. The signature for yielding as evidenced by larger dimple was more predominant in the specimens with no martesite.

Cigada et al. reported [3] similar observations on AISI 316 stainless steel. Their study also confirmed that deformation at 77 K could yield martensitic transformation. They also observed that UTS and YS increased while percentage elongation and percentage reduction in area (%RA) reduced when specimens with increasing quantity of α- phase were tensile tested.

The interesting observation made by Cigada et al. was that the notch sensitivity of the steel increased from 0.28 (for 0% deformation) to 0.84 (for 50% deformation at 77K) with martensitic transformation. This indicated that the material with transformed martensite was sensitive to notch, i.e. transformed martensite caused detrimental effect on the notch sensitivity of the steel.

V. CONCLUSIONS

Deformation of 0.12C-0.5Ti-9Ni-18Cr-2Mn stainless steel at 77K enhanced the kinetics of austenite-martensite transformation. The transformed martensite has adverse reduced impact toughness of steel.

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